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# NANOSTRUCTURED Cu-45 at.% Ni ALLOY PRODUCED BY HEAVY COLD ROLLING

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## ABSTRACT

The microstructure and texture have been studied in a Cu-45 at.% Ni alloy cold-rolled to von Mises strains of 3.5, 4.8 and 5.7. It is shown that similar to other materials the boundary spacing in this alloy decreases with increasing strain and the fraction of high angle boundaries increases. However, in contrast to pure nickel and copper, where the boundary spacing after heavy rolling is greater than 100 nm in any direction, in the Cu-45 at.% Ni alloy cold-rolled to a strain of 5.7 the boundary spacing along the normal direction is approximately 70 nm. At this strain the fraction of HABs measured using the electron backscatter diffraction technique in the Cu-45 at.% Ni alloy is 72%, which is also higher than in aluminum, copper and nickel cold-rolled to a similar strain. This nanostructured Cu-45 at.% Ni alloy is stable against recrystallization during storage at room temperature for 13 months.

## 1. INTRODUCTION

Comparatively cheap Cu-Ni alloys with reduced ferromagnetism are promising substrate materials for coated conductors (Vadlamani, Eickemeyer, Schultz and Holzapfel 2007; Vannozzi, Celentano, Angrisani, Augieri, Ciontea, Colantoni, Galluzzi, Gambardella, Mancini, Petrisor, Rufoloni and Thalmaier 2008). One of the common methods to manufacture such substrates is a rolling assisted biaxially textured substrates (RABiTS) process (Norton, Goyal, Budai, Christen, Kroeger, Specht, He, Saffian, Paranthaman, Klabunde, Lee, Sales and List 1996), in which a very strong cube texture is generated by appropriate annealing after heavy rolling. A number of different Cu-Ni alloys are being tested to obtain substrates with high

fractions of the cube texture and with predominantly low angle grain boundaries. Optimization of their final microstructure and crystallographic texture requires an improved knowledge of the microstructural evolution during rolling and annealing. In the present paper, we investigate the evolution of the microstructure and texture during heavy cold rolling of a Cu-45 at.% Ni alloy. Microstructures and textures in this alloy cold-rolled to different von Mises strains, 3.5-5.7, are characterized using the electron backscatter diffraction (EBSD) technique, and the results obtained are compared with those in other face centered cubic (fcc) materials with medium to high stacking fault energy.

## 2. EXPERIMENTAL

A Cu-45 at.%Ni alloy with an average recrystallized grain size of 10  $\mu\text{m}$  (calculated including annealing twins) was used in the present study. This material was cold-rolled 95%, 98.5% and 99.3%, which corresponds to von Mises strains ( $\epsilon_{\text{VM}}$ ) of 3.5, 4.8 and 5.7, respectively. The deformed samples were examined in the section containing the rolling and normal directions (RD-ND) in a Zeiss Supra-35 field emission gun scanning electron microscope equipped with a Channel 5 EBSD system. EBSD maps for microstructural analysis were collected with a step size of 20-50 nm to cover areas of at least 2000  $\mu\text{m}^2$ . Low and high angle boundaries (LABs and HABs) were defined as those with misorientations 2 – 15° and > 15°, respectively. EBSD texture measurements were made with a step size of 0.5 – 3  $\mu\text{m}$  covering at least 15000  $\mu\text{m}^2$  in each deformed sample. Fractions of different texture components were determined within 15° from corresponding ideal orientations.

## 3. RESULTS

Heavy rolling resulted in a very strong rolling texture with the S {123}<634> component being the dominant texture component at each strain (see Fig. 1). The summed fraction of the three standard rolling texture components (the Copper {112}<111>, S {123}<634> and Brass {110}<112>) reached 91% in the  $\epsilon_{\text{VM}} = 5.7$  sample, which is very similar to the findings of Mishin, Juul Jensen and Hansen (2010) for aluminium cold-rolled to the same strain.

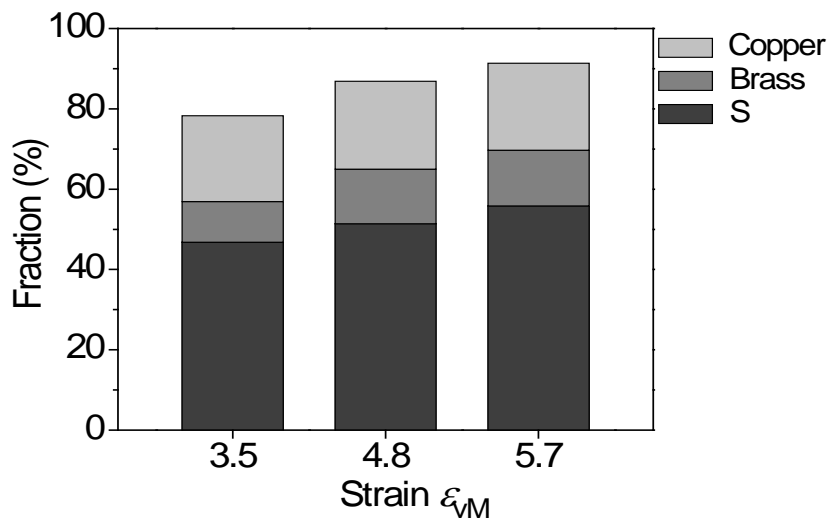


Fig. 1. Volume fractions of rolling texture components as a function of strain.

EBSD maps from Cu-45 at.% Ni reveal deformation structures characteristic of high strain rolling (Hughes and Hansen 2000; Liu, Huang, Lloyd and Hansen 2002; Mishin et al. 2010), comprising lamellar boundaries almost parallel to the rolling plane and microshear bands forming large angles with the rolling plane (see Fig. 2). It is apparent that the tendency to form microshear bands increases with increasing strain (Fig. 2).

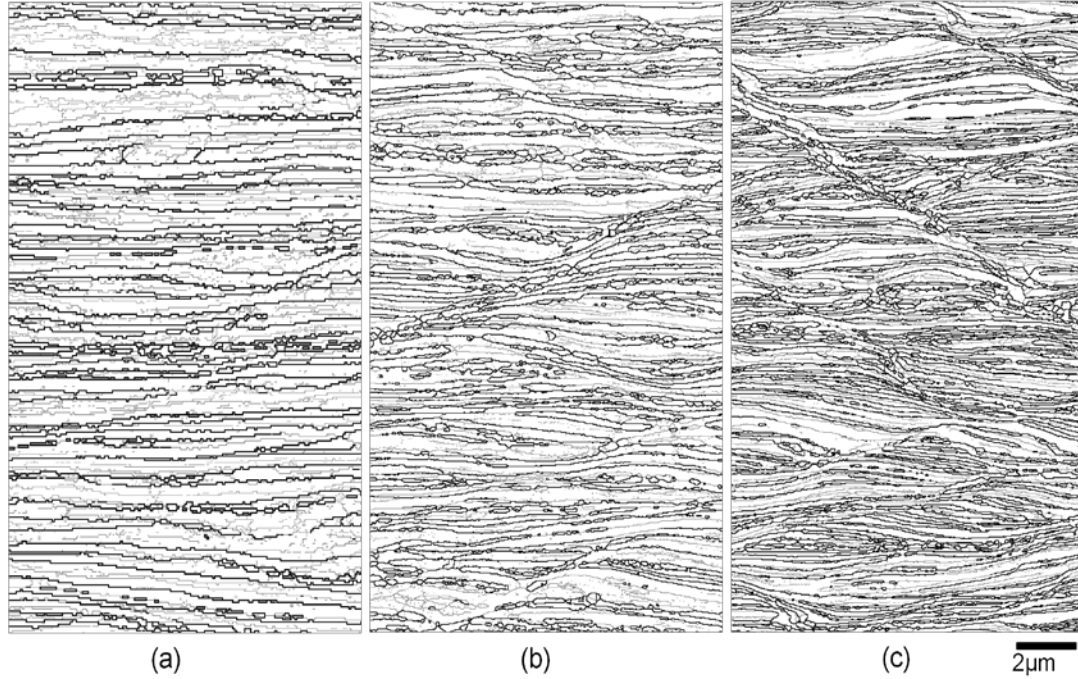


Fig. 2. EBSD maps showing the microstructure in Cu-45 at.% Ni after cold-rolling to different strains: (a)  $\epsilon_{VM} = 3.5$ ; (b)  $\epsilon_{VM} = 4.8$  and (c)  $\epsilon_{VM} = 5.7$ . Grey and black lines show 2-15° and >15° misorientations, respectively.

From analysis of the quantitative data presented in Fig. 3 it is obvious that, as the strain increases, the boundary spacing decreases and the fraction of HABs ( $f_{HAB}$ ) increases. In the  $\epsilon_{VM} = 5.7$  sample, the spacing measured along the ND between all boundary types,  $d_{all}$ , and between HABs,  $d_{HAB}$ , was  $\sim 70$  nm and 100 nm, respectively.

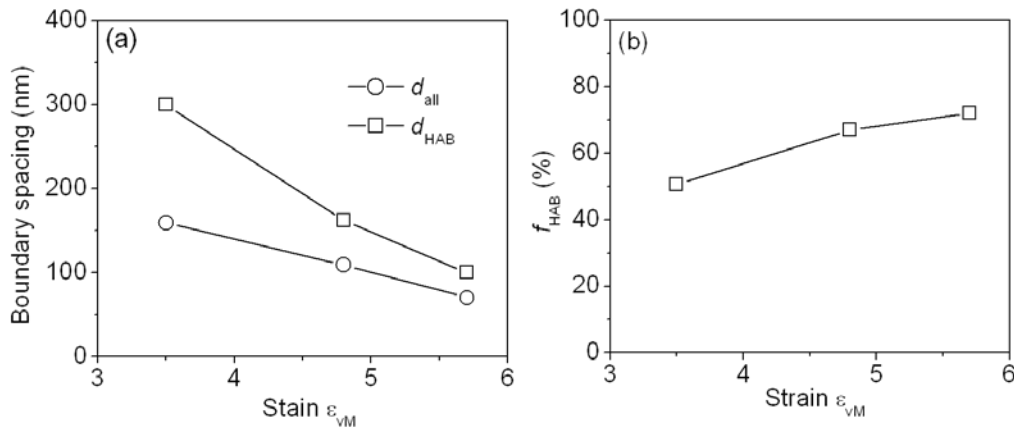


Fig. 3. Microstructural parameters obtained from the EBSD data: (a) boundary spacing measured along the ND between all boundary types ( $d_{all}$ ) and between HABs ( $d_{HAB}$ ); (b) fraction of HABs ( $f_{HAB}$ ).

The  $f_{\text{HAB}}$  in this sample reached 72% (Fig. 3b). Fig.4 demonstrates that the increase in the  $f_{\text{HAB}}$  with increasing strain is due to a reduction in the frequency of  $2 - 5^\circ$  misorientations. Despite a high driving force for recrystallization in this greatly refined material, no signs of recrystallization were observed in the microstructure even after 13 months of room-temperature storage.

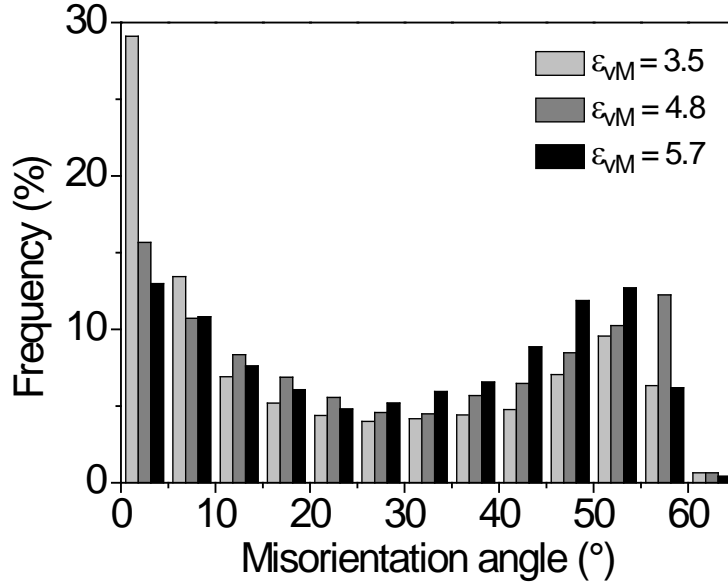


Fig. 4. Distributions of misorientation angles for the Cu-45 at.% Ni alloy cold-rolled to three different strains. Note that since the critical misorientation angle in the EBSD analysis was set to  $2^\circ$  the first bin in the histogram represents  $2 - 5^\circ$  misorientations.

#### 4. DISCUSSION

The microstructural evolution during heavy rolling of the Cu-45 at.% Ni sample appears similar to that previously observed in other fcc materials such as pure Ni and Al (Hughes and Hansen 2000; Liu et al. 2002; Mishin et al. 2010). In agreement with previous reports, the decrease in the lamellae thickness in Cu-45 at.% Ni is accompanied by an increase both in the fraction of rolling texture components and in the  $f_{\text{HAB}}$  (Fig. 1 and Fig. 3). However, compared to AA1050 cold-rolled to a strain of 5.7 (Mishin et al. 2010), the boundary spacing in Cu-45 at.% Ni after the same strain is much finer (290 nm vs 70 nm) and the  $f_{\text{HAB}}$  is larger (50% vs 72%). It is significant that traditional rolling of the Cu-45 at.% Ni alloy is capable of refining the microstructure to the nanoscale level and this refinement is achieved in the microstructure with a high  $f_{\text{HAB}}$ . The greater refinement in the Cu-45 at.% Ni alloy as compared with commercial purity aluminum can be attributed to less dynamic recovery taking place during deformation of this alloy.

The comparison of structural parameters in the Cu-45 at.% Ni alloy after a strain of 4.8 with those in pure Ni previously investigated by the present authors after a similar strain and in pure Cu investigated by Li, Tsuji and Kamikawa (2006) after accumulative roll-bonding (ARB) reveals that the boundary spacings of our alloy are again finer and the  $f_{\text{HAB}}$  is greater than in these pure metals (Table 1).

**Table 1.** Microstructural parameters for deformed Cu-45at.% Ni, Ni and Cu samples measured using the EBSD technique.

Samples	Initial grain size ( $\mu\text{m}$ )	Processing technique	$\varepsilon_{\text{vM}}$	$d_{\text{all}}$ (nm)	$d_{\text{HAB}}$ (nm)	$f_{\text{HAB}}$ (%)
Cu-45 at.% Ni	10	CR	4.8	110	160	67
Ni (99.97%)	13	CR	4.5	140	260	51
Cu (99.99%, Li et al. 2006)	25	ARB	4.8	229	400	54

While the differences in  $d_{\text{HAB}}$  can reflect somewhat different initial grain sizes (see Table 1), the spacing between all boundaries after such a high strain is expected to be less sensitive to the initial grain size. Indeed, the difference in  $d_{\text{all}}$  between the Cu-45 at.% Ni alloy and Ni sample is not significant. However, there is a large difference in  $d_{\text{all}}$  for the rolled Cu-45 at.% Ni alloy and pure Cu sample processed by ARB to a similar strain. It should be noted that the microstructure of heavily deformed pure copper is typically unstable at room temperature (Li et al. 2006; Mishin and Godfrey 2008). Therefore, boundary migration and even partial recrystallization could occur in this metal both between rolling passes and during storage after ARB processing (the latter has been reported by Li et al. 2006). In contrast to pure copper, the microstructure of the Cu-45 at.% Ni alloy is stable against recrystallization during room-temperature storage. The stability of the microstructure at room temperature is important since well-controlled conditions for recrystallization and grain growth are required to obtain a substrate suitable for coated conductors.

## 5 SUMMARY

The microstructure and texture have been studied in a Cu-45 at.% Ni alloy cold-rolled to very high strains,  $\varepsilon_{\text{vM}} = 3.5 - 5.7$ . It has been found that similar to other materials the boundary spacing decreases with increasing strain and the fraction of high angle boundaries increases. After rolling to a von Mises strain of 5.7, the boundary spacing along the normal direction is approximately 70 nm, which is significantly less than in other heavily rolled fcc materials such as Cu, Ni and Al. In contrast to pure copper, the microstructure of the heavily deformed Cu-45 at.% Ni alloy is stable against recrystallization at room temperature. The fact that this alloy is non-ferromagnetic at typical operation temperatures for coated conductors (below 77K) and cheaper than commonly used Ni-based alloys makes the Cu-45 at.% Ni alloy an interesting material for potential applications as a substrate for superconducting tapes. Additional annealing experiments will be conducted to produce a microstructure and texture most suitable for commercial applications.

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